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THE EVALUATION OF LIGNIN PRODUCTS AS BINDERS FOR COKING COAL AND IRON ORE

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1.0 RÉSUMÉ

Des lignines sans soufre obtenues dans les procédés de fabrication de la pâte à papier mis au point par la Stake Technology Ltd. et la Repap Technologies Inc. ont été mises à l'essai en vue de leur utilisation comme liants dans le bouletage du minerai de fer et des fines de charbon à coke de l'Ouest canadien.

Dans le cas du charbon métallurgique Westar-Balmer, l'addition, jusqu'à 0,5 % en poids, de lignine de la Staketech permet de conserver les propriétés de cokéfaction. La liaison des boulettes à cette concentration était tout juste suffisante; les résultats des essais précédents effectués avec de la lignine de la Staketech (gâteau de filtration) non séchée laissent croire qu'une meilleure liaison peut être obtenue. L'addition, à 0,25 et 0,5 % en poids, de lignine de la Repap a fortement réduit les propriétés de cokéfaction du charbon.

Des essais du bouletage préliminaires ont été effectués sur un échantillon de matériau d'alimentation fourni par l'Iron Ore Company of Canada avec les deux lignines, seules et combinées avec de la bentonite. En général, les résultats obtenus avec les deux lignines indiquent

- des améliorations de la rétention d'humidité et de la capacité de bouletage comparables ou supérieures à celles obtenues avec la bentonite;
- une plus grande stabilité des boulettes crues par comparaison à celle obtenue avec la bentonite;
- une résistance équivalente ou supérieure des boulettes sèches par comparaison à celle obtenue avec la bentonite.

Les résistances des boulettes durcies contenant des liants de type lignine étaient inférieures à celles obtenues avec de la bentonite, mais sensiblement supérieures à celles du minerai de fer dont le bouletage a été réalisé sans liant. D'après les résultats d'une étude récente de CANMET, l'addition, au matériau d'alimentation, de liants de type lignine avec du calcaire donnerait aux boulettes durcies des résistances équivalentes ou supérieures à celles obtenues avec la bentonite; de plus, cette façon de procéder réduirait le coût du transport, le volume de laitier et la chaleur de fusion requise.

Une analyse de rentabilité très préliminaire indique que les lignines de la Staketech et de la Repap, ajoutées à raison de 0,5 % environ, pourraient constituer des liants rentables pour le minerai de fer.

Les exigences concernant les travaux futurs sont indiquées.

1.0 SUMMARY

Sulphur-free lignins from pulping processes developed by Stake Technology Ltd. and Repap Technologies Inc. have been tested for use as binders in pelletization of western Canadian coking coal fines and iron ore.

For Westar-Balmer metallurgical coal, the Staketech lignin is compatible with retention of coking properties at up to 0.5 wt % lignin addition. Pellet binding at this dosage was marginal; results of previous tests on undried (filter cake) Staketech lignin suggest that better bonding might be obtainable. The Repap lignin seriously reduced coal coking properties at 0.25 and 0.5 wt %.

Preliminary pelletization tests were done on a pellet feed sample from the Iron Ore Company of Canada with both lignins, alone and combined with bentonite. In general, test results for both lignins indicate:

- moisture retention and ballability improvements similar to or better than bentonite
- improved green ball stability verses bentonite
- equivalent or improved dry pellet strength versus bentonite

Indurated pellet strengths with lignin binders were lower than obtained with bentonite, but substantially better than for iron ore pelletized without binder. Results of a recent CANMET study suggest that lignin binders with limestone addition to pellet feed would produce equivalent or better indurated pellet strength than bentonite; this would also reduce transport cost, slag volume and melting heat requirement.

A very preliminary economic analysis indicates that the Staketech and Repap lignins could be cost effective binders for iron ore at addition levels of approximately 0.5%.

Requirements for further testwork are identified.

2.0 INTRODUCTION

2.1 BACKGROUND

2.1.1 Sulphur Free Lignins

New processes for recovery of cellulose fibre from woodchips are being developed by Stake Technology Limited and Repap Technologies Inc. Both processes have been tested at pilot scale, and commercial applications are either in progress or under active consideration. Both processes produce a sulphur-free lignin by-product for which commercial uses are being sought.

Very briefly, the Stake process technology includes: steam treatment at elevated temperature and pressure, followed by rapid decompression/cooling and extraction of lignin by dilute alkali. After separation of fibre, the lignin by-product is recovered by acidification/liquid-solid separation, and drying.

The Repap process technology involves digestion in liquor containing alcohol and dilute alkali; solubilized lignin is separated from the pulp and recovered by acidification/liquid-solid separation and drying.

Conceptually, both processes may allow lower cost recovery of lignin solids than conventional Kraft or sulphite pulping, because lignin dissolved in the pulping process is precipitated by acidification. Consequently, the amount of water evaporation to generate a dry product is much less than for recovery of soluble lignin from conventional processing. Also, both Kraft and sulphite pulping processes result in a significant content of sulphur in the product lignin. The sulphur contents of these conventional lignins reduce their value for fuel use and - to a large extent - control their physical and chemical properties.

Identification of suitable applications/markets for sulphur-free lignins from the Stake and Repap developmental processes could improve their commercial prospects.

This report presents results of preliminary evaluations of sulphur-free lignins as binders for coking coal and iron ore.

2.1.2 Coking Coal

Coking or "metallurgical" coals produced by western Canadian mines in Alberta and British Columbia contain relatively large amounts of fines (broadly defined as -100 mesh or -150 microns) due to tectonic alteration of coal seams.

High fines content can result in a variety of operational problems including:

- dust loss in transit and/or stockpile,
- poor bin flow properties, and
- operational problems in some types of coke ovens due to pneumatic transport of the fines into oven exhaust flues.

Favourable properties of these coals, particularly high fixed carbon and low sulphur contents, provide market access. However, the proportion of these coals which can be used in coking blends is limited by their low fluidity.

Pelletization is a potentially attractive method of avoiding the problems caused by high fines content of these coals. "Pellets" (roughly spherical agglomerates formed by mixing of fine granular solids with a controlled amount of fluid) behave as large granules and, with a suitable binder to strengthen the pellets, could reduce or prevent most of the problems associated with handling and shipment of the fine fraction of western coals.

Results of previous testwork[1] indicated that the fine fraction of Rocky Mountain coals can be easily pelletized using conventional equipment and techniques. However, tests on a wide range of water-compatible binders did not identify a binder which was compatible with acceptable retention of coking properties of the coal, particularly fluidity.

Petroleum, or "oil" binders are generally compatible with retention of coking properties, and may actually enhance coking behaviour[2]. However, there are major problems associated with use of "oil" binders for wet coal as it is produced from existing coal preparation plants.

In a previous study conducted for the Biomass Energy Development Program[3], unseparated raw product from a biomass conversion process was found to be marginally acceptable as a binder for coking coal. However, commercial development of biomass-to-oil conversion processes does not appear likely in the short term, so products of such processes are not likely to be available for current applications.

In contrast, the processes being developed by Staketech and Repap are likely to be commercialized due to technical and/or economic factors relating to pulp production. Consequently, byproduct lignins from these processes are likely to be available in the near future.

This study presents results of tests using sulphur-free lignins as binders for pelletization of western Canadian coal fines.

2.1.3 Iron Ore

Pelletization of fine iron ore is the largest industrial application of agglomeration technology. Bentonite clay is the predominant binder for this technology, due to its low cost and favourable affects on pelletization behaviour and pellet strength in green, dry and indurated states. However, bentonite added to the pellets represents "acid" gangue which much be fluxed and removed as slag in the steel making process.

Although the amount of bentonite used in pelletization is small, generally on the order of 0.5%, this may represent a significant increase in both basic flux requirement and slag volume in the steel making process.

A wide variety of organic materials have been investigated as partial or complete substitutes for bentonite in pelletization of iron ore. Conceptually, the potential advantages of organic binders include elimination of the bentonite component of acid gangue, associated flux requirement, and melting energy costs. Also, organic binders serve as fuel in the pellet induration process thus reducing the direct fuel cost for induration.

Several recent publications[4,5] have dealt with chemically modified cellulose products which - although costly - appear to provide acceptable green and dry pellet properties at lower addition levels than required for bentonite.

Other recent/current investigations[6] have indicated that particulate organics such as peat moss and sawdust can improve the reducibility of iron ore pellets.

Sulphite lignin, the cheapest commonly available organic binder, has been found not to give acceptable pellet properties.

This study reports results of preliminary tests using sulphur-free lignins as partial and complete replacements for bentonite in pelletization of fine iron ore.

2.2 OBJECTIVES

The "statement of work" (specific terms of reference for this study), is presented as Appendix I.

Briefly, the objective of the study was to perform scoping tests on pelletization of coking coal and iron ore using developmental sulphur-free lignins in order to identify potentially promising applications for these materials.

3.0 MATERIALS AND METHODS

3.1 TEST MATERIALS

3.1.1 Lignin Samples

Lignin samples for this study were provided by Stake Technology Ltd. and Repap Technologies Inc.; both samples were provided as dry, free-flowing powders and were used as supplied.

3.1.2 Coking Coal

The coal used for pelletization testwork was provided by a Westar Mines Ltd. from the Balmer Operation at Sparwood, B.C.; the sample was filter cake from the fine coal cleaning operation.

3.1.3 Iron Ore

A sample of iron ore "pellet feed" was provided by the Iron Ore Company of Canada Ltd., Sept Isle, Quebec; this material was tested as supplied, and also, for control purposes, blended with a sample of bentonite clay used by the Iron Ore Company.

3.2 METHODS

3.2.1 Sample Characterization

Thermogravimetric analysis (TGA) was used to determine moisture, volatile matter, fixed carbon and ash contents of lignin samples. Titrable acidity and water solubility of lignins were determined by wet chemical methods. A preliminary assessment of binder behaviour of the lignins was made by preparation of thin films of dried lignin as supplied, neutralized and treated with alkali to pH 9. Calorific values were determined using a Parr bomb calorimeter.

Elemental analyses of lignin samples were subcontracted to Canadian Microanalytical Inc.

Coal coking properties (free swelling index or "FSI"), fluidity, and dilatation behaviour) were determined by Commercial Testing Limited.

3.2.2 Pelletization

All pelletization tests were done in a laboratory batch test unit fitted with a 20 cm dia. x 30 cm drum; rotation speed and angle of inclination were adjusted on the basis of visual observation to optimize pelletization behaviour for specific mixtures.

Coal samples were pelletized in 200 g batches; iron ore samples were pelletized in 500 g batches.

Pelletized coal samples were screened at 3/8 in. with recycle of oversize. Iron ore pellets were screened at 3/8 in. and 1/4 in. with recycle of over and undersize.

For coal, close control of pellet size was considered unnecessary; for iron ore, a smaller pellet size than used in commercial practice was selected in order to obtain enough pellets from each single batch for a full range of tests.

3.2.3 Pellet Properties

For both coal and iron ore, "green" (moist) pellet strength was assessed by drop testing five selected uniform sized pellets from a height of 45 cm to a concrete surface, and averaging the number of drops to fracture.

Air dry pellets were tested for abrasion resistance by placing ten selected uniform pellets in a Rotap apparatus on a 6 mesh screen and shaking for 10 minutes. Stability was measured as weight percent retained.

For iron ore pellets, air dry, oven dry and indurated pellets were tested for compression strength to failure on an Instron apparatus.

4.0 RESULTS AND DISCUSSION

4.1 LIGNIN PROPERTIES

Tables 1-5 present results of characterization tests on the two lignin samples. The following summary of these data indicates that there are substantial differences in a wide range of properties of the two lignin materials.

	Lignin	
	Stake	Repap
Calorific value (MJ/kg, dry)	23.0	27.7
Titrable acidity to pH 9 (mEq/g, as tested)	6.9	1.0
Moisture (wt % as tested)	14.4	2.8
Fixed carbon (wt %, dry)	22.2	30.0
Carbon (wt %, dry)	56.0	65.5
Oxygen (wt %, dry)	37.8	27.9
Solubility at pH 7 (wt % as tested)	5.1	0.9

Figures 1-4 present results of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) analysis of the two lignin samples.

In general these results appear to indicate that the Repap lignin, either during its extraction or during more extensive drying, has become a more highly condensed product; this is indicated by higher calorific value, higher carbon and fixed carbon contents, lower moisture content and lower solubility except at alkaline pH.

There is a substantial difference in titrable acidity of the materials to both pH 7 and pH 9.

It is considered probable that the analytical properties of the lignins are affected by drying. A previously tested sample of the Stake lignin, containing approximately 50% moisture as received, was found to be virtually completely soluble at pH 9, whereas the current sample was less than 50% soluble at the same pH. Also,

film forming tests with the previous sample of Stake lignin indicated that evaporation of the alkaline solution resulted in a homogenous translucent solid film, whereas evaporation of both the current Stake and Repap lignin samples from alkaline solution resulted in non-homogenous films.

On this basis, it is considered probable that optimization of either lignin product for use as a binder should involve evaluation of both drying conditions (eg. time/temperature), and extent of moisture removal. Observed differences in water solubility and film forming behaviour for filtered and dried samples of Stake lignin suggest that there could be major differences in adhesion properties (ie. green/dry pellet strength), depending on the extent and method of water removal.

Also, as discussed in the following section, pyrolysis properties of the lignins are identified as a potentially significant factor in retention of coal coking properties; volatility of lignin under pyrolysis conditions could well be a function of type and extent of drying.

4.2 PELLETIZATION OF COAL FINES

Results of Rotap stability tests on pelletized coal fines are presented in Table 6.

These data indicate that both lignin products provide significant binding, compared to a control sample of pellets prepared with water only. The Stake lignin sample appears to be a more effective binder with respect to both stability of large (+6 mesh) agglomerates and suppression of dust formed by abrasion in the test. However, even for the sample treated with Stake lignin, abrasion resulted in significant liberation of -100 mesh material. Results of previous tests with completely water soluble sulphite lignin binders gave better results in terms of reduced liberation of -100 mesh material. On this basis, it is considered probable that the binder properties of Stake lignin product for coal could be improved by using material which has not been chemically altered by extensive drying.

Coal pellets were also prepared with both lignins using ammonia to obtain alkaline pH for maximum solubility; results discussed in the following section indicate that ammonia used to dissolve the lignin had a significant effect on coking properties, so the significant test results are considered to be those with lignin binder only.

4.1.1 Coking Properties of Pelletized Coal

Results of free swelling index (FSI) determinations on selected pelletized coal samples are presented in Table 7.

These data indicate that the Stake lignin at 0.5 wt % addition level does not significantly affect FSI whether or not ammonia is used for partial neutralization. (The same amount of ammonia was used in tests with 0.25% lignin as with 0.5%, since it was not anticipated that the ammonia itself might affect coking properties; consequently the reduced free swelling index with Stake lignin (0.25%) plus ammonia is considered to be due to the effect of the ammonia). For the Repap lignin, a significant decrease in FSI was observed both with and without added ammonia.

On this basis, further coking property tests were done on samples without binder (control), and samples with 0.5% Stake lignin.

Results of fluidity and dilatometry tests are presented in Table 8 (see also Appendix II).

The maximum fluidity (10 ddpm) for the control sample illustrates the very low fluidity of this coal. For the lignin treated sample, a lower maximum fluidity (5 ddpm) was observed. Considering the natural low fluidity of the coal, and the limited precision of the fluidity test at such low fluidity levels, it is considered more appropriate to regard the lost fluidity as 5 ddpm absolute rather than 50% of the maximum fluidity. Clearly, further testing would be required to confirm this.

Other fluidity test data are considered to be within the limits of precision of the test procedure.

Similarly, results of the dilatometer tests are considered to indicate the same behaviour for both untreated coal and Stake lignin bonded pellets within the experimental precision of the test procedure.

The principal objective of the scoping tests on coal pelletization was to identify a condition or range of conditions under which acceptable coking properties could be retained; results are considered to indicate that use of the Stake lignin up to 0.5 wt % would be acceptable in terms of retention of coking properties.

Review of the comparative properties of the two lignin samples does not reveal any obvious reason why one should have significantly different properties from the other. However, the graphed results of thermogravimetric analyses (Figures 1 and 2) of the lignin samples indicate that the Stake lignin is pyrolysed more rapidly and at a lower temperature than the Repap lignin. On this basis, it is possible to speculate that pyrolysis of the Stake lignin is largely complete before the onset of coal fusion (i.e. temperature of initial fluidity). For the Repap lignin, the higher pyrolysis temperature and slower rate may result in overlap of the lignin pyrolysis and coal phase change reactions, with consequent chemical and/or physical interference in the latter.

In any event, the results indicate that under identified conditions the Stake lignin product may be acceptable for use as a binder on sensitive, low fluidity coals.

Confirmation tests on a range of fine coal products, preferably including tests on samples with significantly higher natural fluidity, would be required to adequately document this conclusion. However, considering the uniformly negative results obtained in previous tests with lignin and other biomass derived binders, the present result is considered positive.

Further testing should include evaluation of the Stake lignin product without thermal drying (i.e. as filter cake), since results of previous tests on such material indicated significantly better film formation which suggests better binding properties.

4.2 IRON ORE PELLETIZATION

Qualitatively, both lignins improved the "ballability" of the iron ore concentrate to about the same (visual) extent as bentonite.

Results of stability tests and crushing strength tests on iron ore pellets with selected binders are presented in Tables 9 and 10.

In considering these results, it should be noted that the pelletization conditions in the small test drum are radically different from those prevailing in commercial sized equipment. Thus, results from this test program with various binders can be compared internally with reasonable assurance of validity, since all pellets were prepared under essentially the same conditions of compression and retention time. However, the higher compressions and longer retention times in commercial pelletization operations, as well as different time/temperature conditions in the induration stage, make comparisons of lab scale results with commercial practice somewhat questionable.

With this reservation, drop shatter test results (Table 9) indicate marginally adequate drop shatter behaviour for control pellets prepared with 0.5% and 1% bentonite (average four drops in both cases). Significantly better results were obtained on pellets formed with lignin only, especially the Repap lignin at 2 wt % addition level. Also the combined use of lignin (2%) and bentonite (0.5%) provided significantly improved drop stability. It should be noted that, prior to drop-testing, pellets were dried in the rotating drum to the point at which no moisture stain was visible on the drum surface. Moisture contents of "green" pellets (Table 9) clearly indicate the water immobilization properties of both lignins; this property is generally regarded as a useful index of binder quality with respect to green strength. Also, drop test results are generally better at higher (i.e. as-formed) moisture contents, so results presented in Table 9 may underestimate drop stability.

Rotap stability test results on air dry and oven dry pellets (Table 7) generally indicate that pellets prepared with lignin only had superior properties to pellets

prepared with bentonite binder only. This is particularly evident in the results of tests on oven dried samples.

The results suggest only a slight difference in air dry and oven dry stability between lignin bonded pellets prepared with and without addition of ammonia. Based on the observed superior film forming characteristics of lignins prepared by drying alkaline solutions, it was expected that these samples would display superior binding characteristics. The absence of any large affect on binding properties in tests with added ammonia suggest that the acidic functional groups of the lignins may be bonding to the iron ore surfaces, thus (in affect) being neutralized in situ.

Generally the best Rotap stability results were obtained with a combination of 2% lignin and 0.5% bentonite as binder. The relatively low air dry stability of pellets with Repap lignin and bentonite is anomalous in this respect, as are crushing strength test results for the same sample (see Table 10). However, oven dried pellets from the same batch showed the best stability observed for any combination of binders.

Air dry and oven dry pellets formed from the iron ore sample without binder additive showed negligible stability in the Rotap test.

For indurated pellets, the best Rotap stabilities were observed for bentonite (1%) or bentonite lignin mixtures. This is an expected result, since the lignin is completely burned out under induration conditions and provides no residual strength. However, it is interesting to note that pellets prepared with only lignin binder were much more stable than those containing no binder after induration and removal of the organic phase. This observation suggests that both lignins have an affect similar to that of bentonite with respect to improvement of pellet formation (i.e. compaction, spherical shape, and freedom from cracks or other defects).

Results of crushing strength tests (Table 10) generally parallel those of the Rotap stability test.

In both the air dry and oven dry condition, strengths of pellets formed with lignin binder ranged from equal to significantly higher than those formed with 0.5% bentonite.

As previously noted, the combination of Repap lignin (2%) with 0.5% bentonite showed an anamolous low strength in the air dry condition. However, this sample showed the highest observed crushing strength in both the oven dry and calcined condition.

Pellets formed from iron ore without binder showed negligible crushing strength in the air dry and oven dry conditions. Crushing strength for indurated ore pellets varied wildly (0.5 to approx. 20 kg load). Visual observation of these pellets before crushing indicated that the generally poor crushing strength was due to poor pellet shape and/or presence of cracks.

For oven dried pellets, observed average crushing strength values were generally below the 4.5 kg level specified by the International Iron and Steel Organization (ISO) for acceptable pellets. This is considered to be due to the small size of pellets produced (as previously noted, to permit a full range of tests on a single batch of pellets), as well as the low compression loading of pellets in the small pelletization unit. Internal comparison of results clearly indicates that improved dry pellet properties can be obtained with lignin binders only or with lignin-bentonite combinations, compared to properties obtainable with bentonite binder only.

For indurated pellets with bentonite binder at 0.5 and 1%, results were better than those obtained with lignin binder only. This is an expected result based on enhanced high temperature bonding between bentonite and iron ore particles due to ferrite and slag formation. Also, the calcination temperature of 1100°C (dictated by the maximum temperature of the available muffle furnace) was not high enough for development of maximum pellet strength with or without bentonite present.

Results of a recent CANMET study[4] (see also Appendix III), clearly show a strong dependence of indurated pellet crushing strength on induration temperature and soaking time at maximum temperature.

4.2.1 Commercial Potential of Organic Binders for Iron Ore Pellets

Successful commercial use of organic binders for pelletization of iron ore will require a combination of low operating cost and acceptable performance in terms of pellet properties in the green, dry and indurated conditions.

Due to the preliminary nature of this study, it is necessary to make some extrapolations of the results in order to assess the commercial potential of sulphur-free lignins as binders for iron ore pellets. Results of the current study indicate:

- the green and dry condition properties of iron ore pellets prepared with sulphur-free lignin binders are generally equivalent to or better than those of pellets prepared with bentonite only, and

- properties of indurated pellets prepared with organic binders only are marginally inferior to those of pellets prepared with bentonite.

Results of the 1987 CANMET study[4] (see also Appendix III), show that the strength of indurated pellets prepared with organic binder and without bentonite can be increased to ISO acceptable levels by addition of limestone to the pellet mix. This is a very important observation in relation to the general concept of using organic binders, since - instead of adding acidic (silica plus alumina) material to the pellet to obtain indurated strength - basic material is being added, which moves the overall pellet composition towards being self fluxing. This has important implications in relation to pellet reduction in a blast furnace. In effect, addition of limestone to obtain acceptable indurated pellet strength utilizes the natural components of iron ore to develop ferrite and calcium silicate (slag) bonding in the indurated pellet rather than adding extraneous acid gangue (i.e. bentonite).

The energy requirement for pellet induration with limestone addition will be increased due to the endothermic decomposition of limestone to lime. Conceptually, however, the fuel value of an organic binder (or even the fixed carbon residue) would be more than sufficient to complete this reaction.

It should be noted that addition of limestone to a pellet mix containing lignin with acidic properties will result in limestone decomposition and CO_2 evolution up to a pH of about 7. The effect of this reaction on the binding properties of Stake and Repap lignins has not been tested. Data on titrable acidity presented in Table 2 suggest that this would be a more potentially important consideration for the Stake lignin than for the Repap lignin. In either case, since little of the test material is soluble at pH 7 it is not considered likely that the limestone reaction would significantly alter binding properties.

The combination of current test results on low cost organic (lignin) binders and the results of CANMET investigations on organic binders with limestone addition is considered to form reasonable technical justification for further experimental work in evaluation of the two sulphur-free lignins for use in iron ore pelletization.

If acceptable pellet strength properties can be proven by further testwork, then use of organic binders has a further potential for improving iron ore pellet properties in the area of reducibility. Reports of recent studies in Canada, Japan and elsewhere[6,7,8] indicate that reducibility (the rate at which iron oxide reduction occurs under constant conditions of temperature and reducing gas) can be substantially improved by addition of peat moss, sawdust and other organic materials to pellet feed. Pyrolysis and combustion of these materials during pellet induration results in porosity which allows the reducing gas access to the interior of the pellet, leading to homogenous rather than topochemical reduction.

4.2.1.1 Cost Factors

The recent CANMET study[4] investigated chemically modified celluloses and guar gum; there are also numerous reports regarding use of "PERIDUR" (a commercial cellulose derived binder). Examination of material cost data presented in Table 11 clearly indicates that such materials must be used at very low levels of addition in order to be cost-competitive with bentonite.

Since neither of the two lignins used in the current study are commercially produced, there are no reliable cost data for these materials. However, the current price for sulphite lignin is considered a reasonable first approximation. Conceptually, since both the Stake and Repap process lignins are recovered by precipitation, the evaporation costs associated with production could be significantly lower resulting in a lower final product cost. However, even if the developmental lignins are assumed to be cost-competitive with sulphite lignin, there is a factor of approximately 10-15 difference between the cost of lignin and previously investigated cellulose or gum binders. On this basis, use of lignin at a higher dosage level could be justified.

Economic analysis of lignin or other organic binders compared to bentonite is complicated by the numerous site specific considerations relating to binder use at a particular pellet producing facility. The following analysis (with noted assumptions) indicates that the direct cost of lignin binder at the same dosage as for bentonite would be approximately 50% higher.

NET COSTS OF BINDERS

Lignin		
(Assumed) dlvd cost	$ 200	/tonne, dry basis (equivalent to sulphite lignin)
Subtract fuel equivalent cost ($2/GJ for sulphur free heat units)	$ 55	/tonne, dry basis
Net cost	$ 145	/tonne
Potential benefits	+ enhanced reducibility + reduced slag volume	
Bentonite		
(Assumed) dlvd cost	$100/tonne	
Liabilities	- transport of acid gangue to blast furnace - flux cost - melting energy - slag disposal	

However, there are significant potential additional benefits to use of lignin (or other organic binder) in relation to pellet reducibility, reduced slag volume and other factors. The negative features associated with bentonite use (transport, flux, melting energy, slag disposal and other items) are all site specific.

In general, it appears that the potential benefits associated with use of lignin binders could balance or exceed the cost differential provided that lignin binders can be proven effective at about the same dosage level (generally approx. 0.5%) as required for bentonite.

4.3.2 Direction of Future Investigations

The current results, with noted assumptions and extrapolations, suggest that sulphur-free lignins could be cost effective as iron ore pellet binders. Critical technical areas for future, more detailed investigation include:

- confirmation that lignin provides acceptable green and dry strength at dosages of approx. 0.5%, using test equipment which provides pelletization conditions equivalent to industrial seale equipment,
- confirmation that lignin binders are compatible with limestone addition to the pellet mix (as shown by CANMET to be required for acceptable indurated strength in previous work on more exotic organic binders),
- confirmation of anticipated enhanced pellet reducibility, and
- demonstration in larger test apparatus that lignin provides the same improvement in "ballability" as bentonite. (N.B. This scale of testing requires facilities available at CANMET and in producers test facilities).

Subject to satisfactory results in these areas, it appears likely that lignins of the types investigated in this study could be cost competitive and technically acceptable in commercial pelletization of iron ore.

Economic feasibility of lignins as iron ore pellet binders depends on:

- availability of lignin in suitable quantities,
- delivered price, and
- compatibility of lignin with existing material storage and handling systems.

Clearly, availability of suitable lignin(s) is contingent on commercialization of one or both of the Repap and Staketech processes. Delivered price will depend on several factors including:

- pulp plant location
- alternate markets for lignin byproducts

Economic justification of further testwork at larger scale and followed up by pellet property analyses will require some degree of assurance that suitable quantities of lignin could be made available at prices competitive with sulphite lignin.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 PELLETIZATION OF COKING COAL

Results of this study indicate that lignin derived from the Stake Technology pulping process can be used as a pelletizing binder for sensitive, low fluidity coking coals. Further testwork would be required to document this to a level of certainty required for coal marketing, but the present results are considered positive.

Use of pelletizing technology for coal fines will require a different approach to drying than the fluid bed technique used at most currently operating mines. For this reason, pelletization technology is more likely to be adopted by a new coking coal mining operation.

5.2 IRON ORE PELLETIZATION

Results of this study indicate that the Stake Technology and Repap lignins can provide improved green pellet properties compared to bentonite. Subject to experimental confirmation (see Section 4.2.2) that acceptable properties can be obtained with lignins at (about) 0.5% level, with limestone addition for indurated strength, site specific cost-benefit studies would be appropriate.

ACKNOWLEDGEMENTS

Financial support for this study from the Biomass Energy Development Program is gratefully acknowledged, as is assistance from Mr. Ed Hogan in all phases of the project.

Assistance in the form of test samples and information was also supplied by Stake Technology Ltd., Repap Technologies Inc., the Iron Ore Company of Canada, and Westar Coal Ltd.

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4. Kuriakose, A.K., and D.H. Quon, Pelletization of Iron Ore by Partial or Full Substitution of Bentonite by Organic Binders, CANMET Mineral Sciences Laboratories, July, 1987.

5. Sastry, K.V.S. and A. Negm, Role of Binders During Pelletization - Pellet Growth and Strength Aspects, 4th International Symposium on Agglomeration; C.E. Capes, ed., Toronto, 1985.

6. Panigrahy, S.C. et. al., Substitution of Bentonite with Peat Moss and its Effect on Pellet Properties, 4th International Symposium on Agglomeration; C.E. Capes, ed., Toronto, 1985.

7. Appleby, J., and G. Shaw, Carbonaceous Additives in the Pelletizing Process, 4th International Symposium on Agglomeration; C.E. Capes, ed., Toronto, 1985.

8. Nisheda, I., T. Uenaka and I. Mizuguchi, Blast Furnace Performance Using Various New Pellets at Kakogowa Works, HF80 Conference on Operation of the Blast Furnace - Theory and Practice, Arles, France, June, 1980.

TABLE 1

CALORIFIC VALUES OF LIGNINS

Sample		As Tested		Dry Basis	
		Btu/lb	MJ/kg	Btu/lb	MJ/kg
Stake	1	8,643			
	2	8,316			
	Mean	8,480	19.7	9,906	23.0
Repap	1	11,568			
	2	11,587			
	Mean	11,576	26.9	11,909	27.7

TABLE 2

TITRABLE ACIDITY OF LIGNINS*

		milliequivalent/g (as tested)		Dry Basis Average Gram Molecular Weight per Titrable Equivalent	
		Stake	Repap	Stake	Repap
to pH	7.0	2.3	0.3	435	3000
	9.0	6.9	1.0	145	1000

* natural pH values for 10% slurries:

Stake 3.7
Repap 4.1

TABLE 3

PROXIMATE ANALYSES OF LIGNINS

	As Tested wt %				Dry Basis wt %		
Sample	Moisture	Volatile Matter	Fixed Carbon	Ash	Volatile Matter	Fixed Carbon	Ash
Stake	14.4	64.9	18.6	0.1	77.6	22.2	0.1
Repap	2.8	68.5	29.3	0	70.0	30.0	0

TABLE 4

ELEMENTAL ANALYSES OF LIGNINS

	As Tested, wt %				Normalized*, wt%			
Sample	C	H	N	O	C	H	N	O
Stake	47.7	5.2	0.092	32.2	56.0	6.1	0.11	37.8
Repap	64.7	6.4	0.12	27.6	65.5	6.5	0.12	27.9

* Correction for moisture content.

TABLE 5

WATER SOLUBILITY OF LIGNINS

	wt % soluble at pH		
Sample	Natural pH*	7.0	9.0
Stake	1.7	5.1	42.7
Repap	0.4	0.9	37.4

* see Table 2

TABLE 6

RESULTS OF STABILITY TESTS* ON BALMER COAL FINES PELLETIZED WITH SELECTED BINDERS

Binder	Wt % retained	
	6 mesh	Pan (-100 mesh)
None	5	42
Stake Lignin, 0.5%	76	12
Repap Lignin, 0.5%	62	18

* 10 g air dry pellets; Rotap 10 minutes

TABLE 7

FREE SWELLING INDICES OF BALMER COAL WITH SELECTED PELLETIZING ADDITIVES

Additive(s)	FSI	
	Initial	Confirmation
None* (control)	6.5	7.0
Ammonia	4	
Ammonia + Stake Lignin, 0.5%	6.5	
Ammonia + Stake Lignin, 0.25%	4	
Ammonia + Repap Lignin, 0.5%	4.5	
Ammonia + Repap Lignin, 0.25%	3	
Stake Lignin, 0.5%		6.5
Repap Lignin, 0.5%		4.5

* See text for pelletization conditions

TABLE 8

EFFECT OF STAKE LIGNIN ON PLASTIC PROPERTIES OF BALMER COAL

		Test Sample	
		Control*	Coal + 0.5 wt % Lignin
Gieseler Plastometer			
Max. Fluidity DDPM **		11	6
Max. Fluidity	°C	465	466
Initial Softening	°C	433	436
Solidification	°C	487	485
Fluid Range	C°	54	49
Audibert-Arnu Dilatometer			
Max. Contraction	%	25	23
Max. Dilation	%	-14	-15
Initial Softening	°C	394	403
Initial Dilation	°C	459	458
Max. Dilation	°C	480	486
Free Swelling Index		7	6.5

* Coal pelletizing with water only

** Dial divisions per minute at 40 g torque

TABLE 9

COMPARATIVE PROPERTIES OF IRON ORE PELLETS WITH SELECTED BINDERS

Binder & Dosage	Green Pellets		Stability* (Dry & Calcined Pellets)		
	H_2O	Drop No.	Air Dry	Oven Dry	Indurated**
None (control)	4.5	3	0.5	ND	77.2
Stake Lignin, 1% + NH_3	8.5	3-4	81.0	83.7	91.4
Repap Lignin, 1% + NH_3	8.4	3-4	60.9	78.8	90.8
Stake Lignin, 2% + NH_3	8.5	7	74.3	86.8	80.7
Repap Lignin, 2% + NH_3	8.6	7	91.3	90.7	84.0
Bentonite, 1%	5.7	4	69.9	78.1	96.9
Bentonite, 0.5%	5.5	4	64.6	69.5	98.8
Stake Lignin, 2% with 0.5% Bentonite	8.3	10	94.1	89.4*** 93.9***	98.7
Repap Lignin, 2% with 0.5% Bentonite	8.5	7-8	61.3	96.3	96.1
Stake Lignin, 2%	8.5	5	87.9	83.1	77.5
Repap Lignin, 2%	8.3	8	91.4	86.6	90.9

* wt % + 6 mesh material after 10 minutes on Ro-Tap

** 10 minutes at 1100°C

*** duplicate tests indicate precision

TABLE 10

CRUSHING STRENGTHS OF DRIED AND INDURATED IRON ORE PELLETS WITH SELECTED BINDERS

Binder(s) & Dosage	Crushing Strength (kg)					
	Air Dry		Oven Dry		Indurated*	
	Mean	(Std. Dev.)	Mean	(Std. Dev.)	Mean	(Std. Dev.)
Stake Lignin, 1% + NH_3	2.46	(0.66)	3.04	(0.93)	13.4	(3.0)
Repap Lignin, 1% + NH_3	1.95	(0.20)	2.55	(0.46)	17.95	(5.1)
Stake Lignin, 2% + NH_3	2.5	(0.41)	2.96	(1.3)	17.15	(6.2)
Repap Lignin, 2% + NH_3	3.7	(0.70)	3.04	(0.48)	17.15	(6.4)
Bentonite, 1%	3.17	(0.5)	2.5	(0.66)	26.5	(7.0)
Bentonite, 0.5%	2.3	(0.28)	2.3	(0.42)	24.0	(5.7)
Stake Lignin, 2% with 0.5% Bentonite	3.9	(0.87)	4.9	(1.2)	18.7	(7.5)
Repap Lignin, 2% with 0.5% Bentonite	1.6	(0.18)	16.9	(4.0)	30.9	(5.6)
Stake Lignin, 2%	2.3	(0.41)	2.9	(0.47)	18.5	(5.3)
Repap Lignin, 2%	3.1	(0.57)	2.8	(0.51)	14.8	(5.8)

* 10 minutes at 1100°C

TABLE 11

COMPARATIVE COST DATA

FOR VARIOUS PELLET BINDERS

Binder	Delivered Cost* ($Can./tonne)	Notes
Bentonite	100	Silica and alumina require basic fluxes.
Sulphite Lignin	200	Ammonia salt, dry weight basis, fuel value approx. 50% of oil
Carboxymethyl Cellulose	2000	No residue, fuel value less than lignin on dry wt basis
Guar Gum	3000	No residue, fuel value 75-80% of oil on weight basis

* Approximate, based on Chemical Marketing Reporter, March, 1989; $(Can.)1.00 = $(U.S.)0.84

FIGURE 1

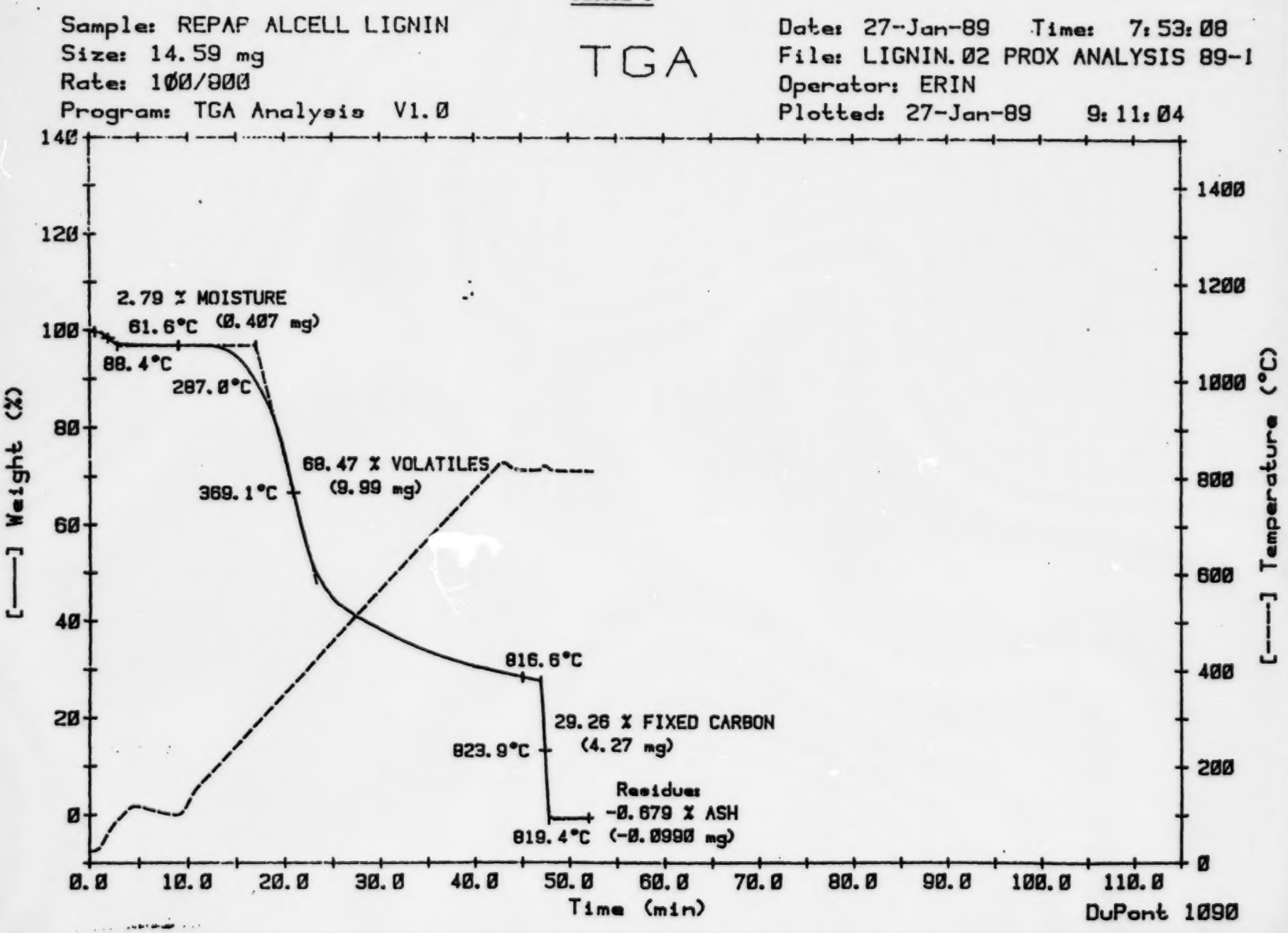

Sample: REPAP ALCELL LIGNIN
Size: 14.59 mg
Rate: 100/800
Program: TGA Analysis V1.0
TGA
Date: 27-Jan-89 Time: 7:53:08
File: LIGNIN.02 PROX ANALYSIS 89-1
Operator: ERIN
Plotted: 27-Jan-89 9:11:04
2.79 % MOISTURE
61.6°C (0.407 mg)
88.4°C
287.0°C
68.47 % VOLATILES
(9.99 mg)
369.1°C
816.6°C
29.26 % FIXED CARBON
(4.27 mg)
823.9°C
Residue:
-0.679 % ASH
819.4°C (-0.0990 mg)
[——] Weight (%)
[----] Temperature (°C)
Time (min)
DuPont 1090

FIGURE 2

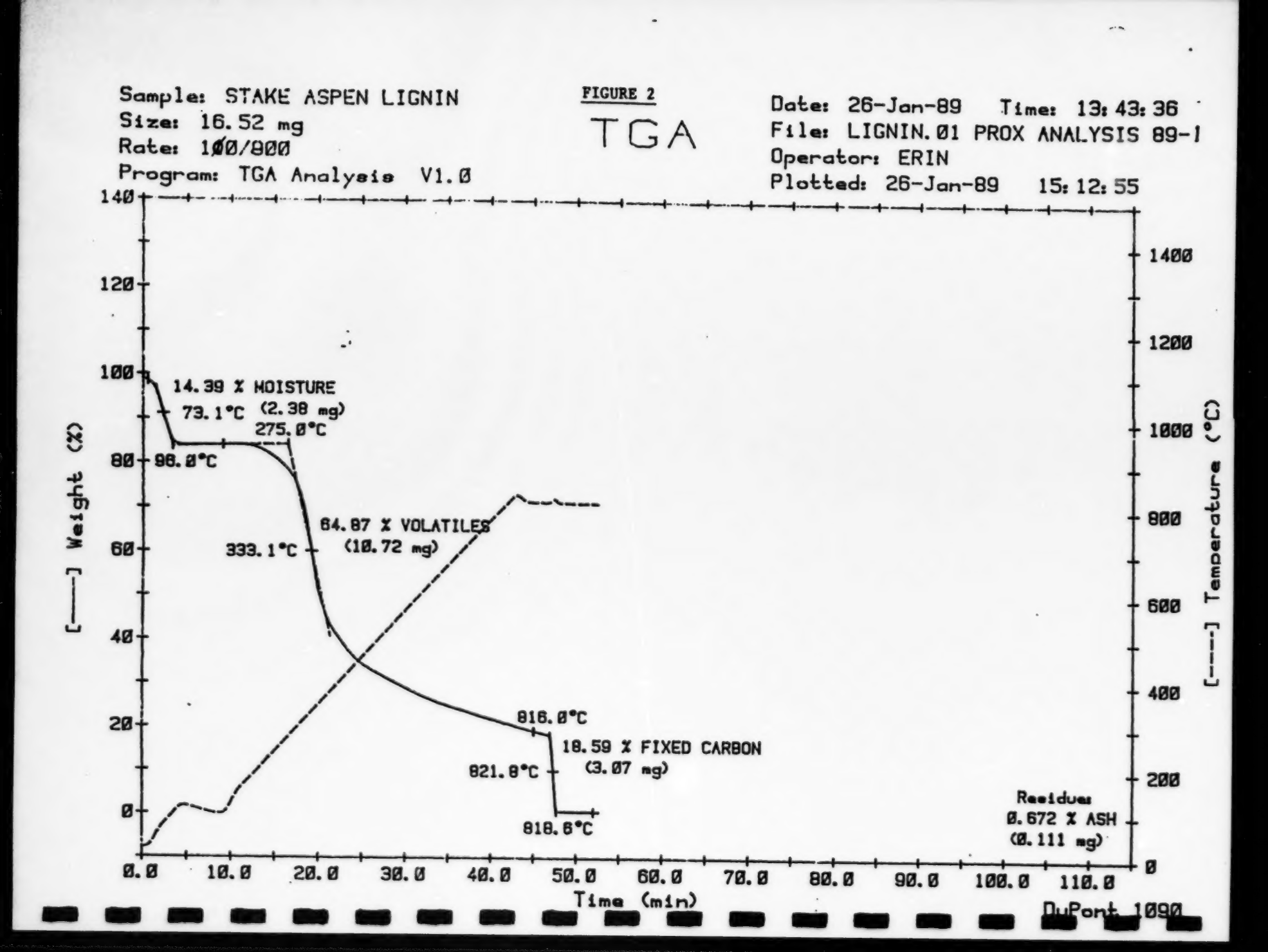

TGA
Sample: STAKE ASPEN LIGNIN
Size: 16.52 mg
Rate: 10Ø/8ØØ
Program: TGA Analysis V1.Ø
Date: 26-Jan-89 Time: 13:43:36
File: LIGNIN.Ø1 PROX ANALYSIS 89-1
Operator: ERIN
Plotted: 26-Jan-89 15:12:55
14.39 % MOISTURE
73.1°C (2.38 mg)
275.Ø°C
96.Ø°C
64.87 % VOLATILES
(10.72 mg)
333.1°C
816.Ø°C
18.59 % FIXED CARBON
(3.07 mg)
821.8°C
818.6°C
Residue
Ø.672 % ASH
(Ø.111 mg)
[——] Weight (%)
[----] Temperature (°C)
Time (min)
DuPont 1090

FIGURE 3

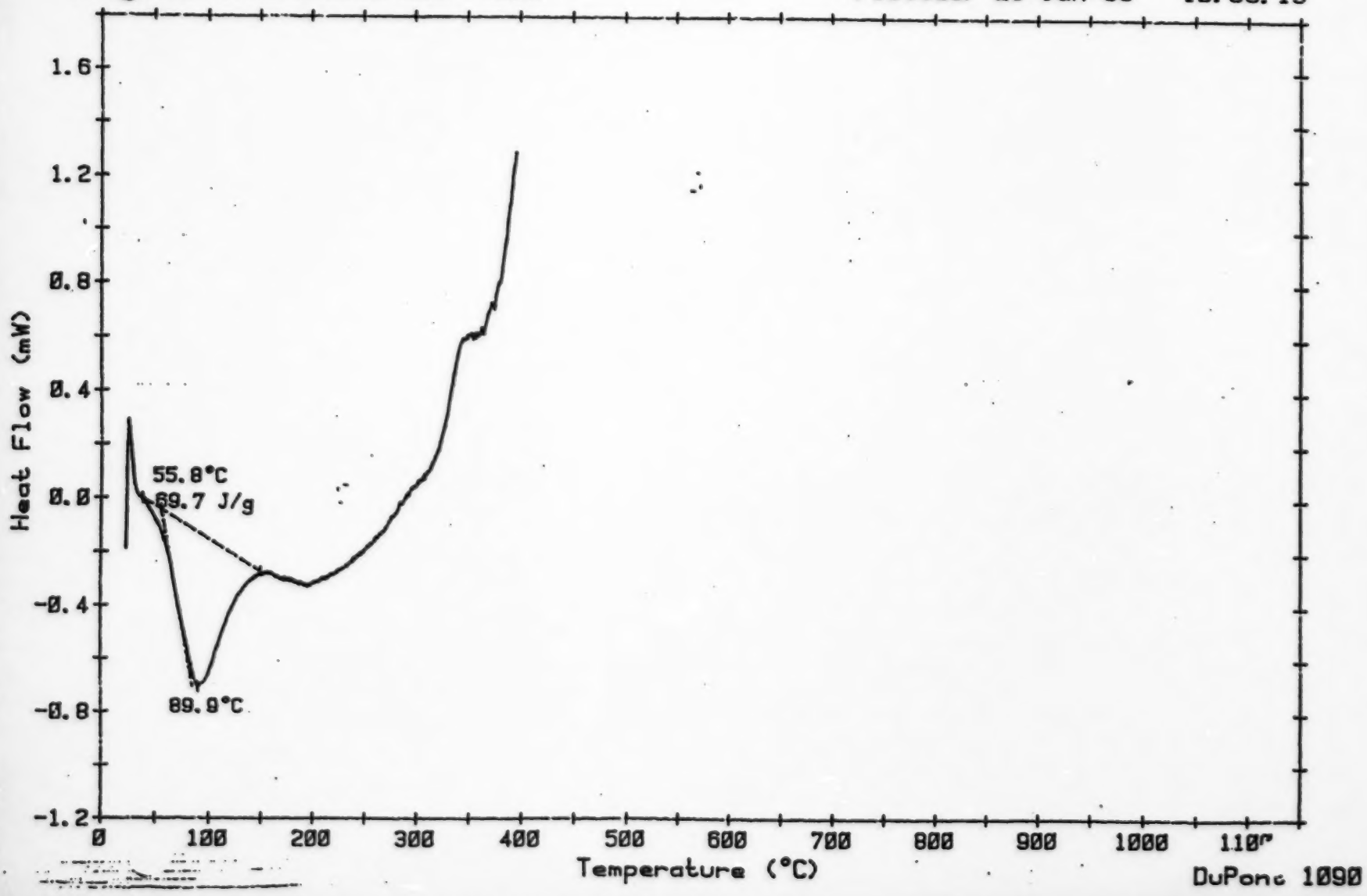
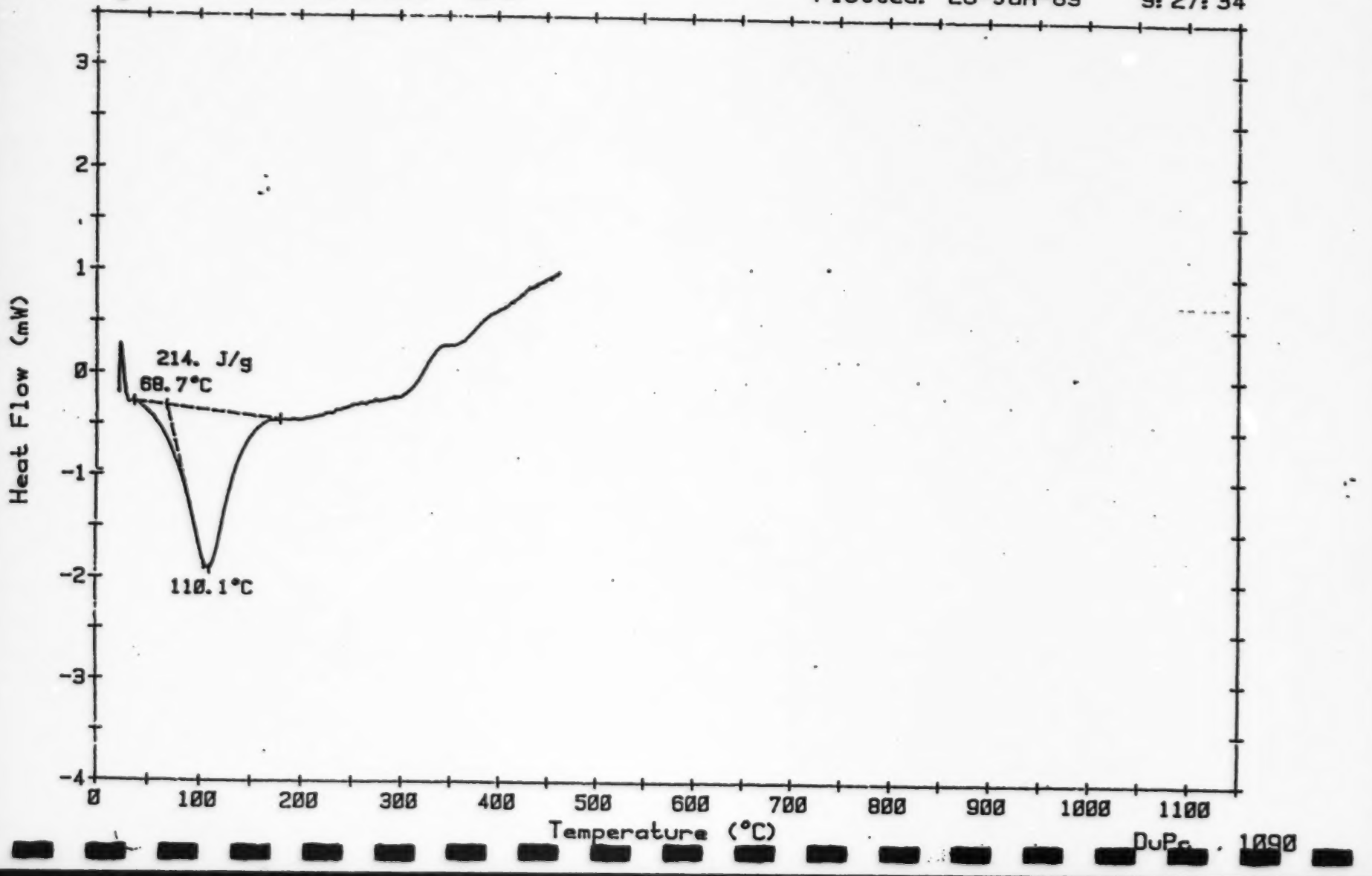

Sample: RA LIGNIN
Size: 2.92 MG
Rate: 10 DEG/MIN IN N2
Program: Interactive DSC V2.0
DSC
Date: 26-Jan-89 Time: 10:03:33
File: LIGNIN.02 DSC MISC 87-1
Operator: ERIN
Plotted: 26-Jan-89 10:56:13
1.6
1.2
0.8
0.4
0.0
-0.4
-0.8
-1.2
Heat Flow (mW)
55.8°C
69.7 J/g
89.9°C
0
100
200
300
400
500
600
700
800
900
1000
Temperature (°C)
DuPont 1090

FIGURE 4


Sample: STAKE LIGNIN
Size: 2.67 MG
Rate: 10 DEG/MIN IN N2
Program: Interactive DSC V2.0
DSC
Date: 26-Jan-89 Time: 8:25:55
File: LIGNIN.01 DSC MISC 87-1
Operator: ERIN
Plotted: 26-Jan-89 9:27:34
214. J/g
68.7°C
110.1°C
Heat Flow (mW)
3
2
1
0
-1
-2
-3
-4
0
100
200
300
400
500
600
700
800
900
1000
1100
Temperature (°C)
DuPo 1090

APPENDIX I

TERMS OF REFERENCE

APPENDIX "A" - STATEMENT OF WORK
****

Requirement: The evaluation of two sulphur-free lignins produced from bioconversion processes for use in agglomeration of coking coal and iron ore as follows:

A. TASKS
*****

1. Agglomeration of Coking Coal
Disc pelletize coal-lignin with/without modifiers (pH adjustment, viscosity adjustment, oil emlusion) to identify acceptable dosage and conditions for dust free product.

Measure free swelling index (FSI) and coke strength of products, using water-only pellets as controls for oxidation effects.

Investigate post-pelletization treatment with low levels of hydrocarbon additives as a possible approach to improved coking properties.

2. Pelletization of Iron Ore
Pelletize hematite with lignin modifiers under a range of conditions and dosage levels to identify conditions for acceptable green/dry strength as indicated by drop-shatter and compression tests.

Conduct control pelletization tests with bentonite and other binders to allow pellet qualify comparison.

Compare lignin and control pellets in a lab scale pot-grate and determine comparative strengths/stabilities of finished pellets.

Note: For both coking coal and iron ore, emphasis will be placed on testing a range of conditions to identify-if possible - a range of dosage and procedures which give acceptable product properties. It is anticipated that effort will be split about 50-50 between coal and iron ore, but if one material shows particular promise, flexibility to shift emphasis is desirable.

It is not expected that optimized conditions will be identified for either product; rather, a "yes...maybe...no" assessment of feasibility will be obtained.

APPENDIX II

COMMERCIAL TESTING REPORTS

COMMERCIAL TESTING & ENGINEERING CO.
1707 FRANKLIN STREET, VANCOUVER, B.C. V6L 1P9 • TEL. (604) 253-2988, FAX (604) 254-4323
DIVISION OF
CT & E TESTING CORPORATION

CERTIFICATE OF ANALYSIS

March 2, 1989

B.C. RESEARCH
3650 Wesbrook Mall
Vancouver, B.C.
V6S 2L2

SAMPLE I.D.: Project No. 2 51 836

P.O. # 18760

Report No.: 04-23809-6

Free Swelling Index

Sample #	FSI	
1	6.5	C.W
2	4	C.W. NH_3
3	6.5	C.W 0.5 [illegible] NH_3
4	4.5	.5 [illegible] NH_3
5	4	.25 [illegible]
6	3	[illegible]

M. Byrne
COMMERCIAL TESTING & ENGINEERING CO

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DIVISION OF
CT & E TESTING CORPORATION

CERTIFICATE OF ANALYSIS

B.C. RESEARCH
3650 Wesbrook Mall
Vancouver, B.C.
V6S 2L2

March 8, 1989

SAMPLE I.D.: Project # 2-51-836

Requisition # R 27434 No P.O.# supplied.

Report No.: 64-23015-3

Free Swelling Index

Sample I.D.	FSI
Coal-Water Coat 2-51-836	7
Stake 0.5% 2-51-836	6.5
Alcell 0.5% 2-51-836	4.5

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CT & E TESTING CORPORATION

CERTIFICATE OF ANALYSIS

B.C. Research
3650 Wesbrook Mall
Vancouver, B.C.
V6S 2L2

March 17, 1989

Sample I.D.: Coal Water Pellets - 251-86

P.O. # 18853

Report No.: 64-23833-2

Plastic Properties of Coal

Gieseler Plastometer

MAXIMUM FLUIDITY, D.D.P.M.†		11
TEMPERATURE AT MAXIMUM FLUIDITY	°C	465
INITIAL SOFTENING TEMPERATURE (1DDPM)	°C	433
SOLIDIFICATION TEMPERATURE	°C	487
TEMPERATURE RANGE	°C	54

Audibort - Arnu Dilatometer

% MAXIMUM CONTRACTION		25
% MAXIMUM DILATATION		-14
INITIAL SOFTENING TEMPERATURE	°C	394
INITIAL DILATATION TEMPERATURE	°C	459
MAXIMUM DILATATION TEMPERATURE	°C	480

† 1 dial division per minute at 40 gram torque

COMMERCIAL TESTING & ENGINEERING CO

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APPENDIX III

EFFECTS OF INDURATION TIME/TEMPERATURE AND CALCIUM CARBONATE ADDITION ON INDURATED STRENGTH OF IRON ORE PELLETS

SOURCE:

Kuriakose, A.K. and D.H. Quon
Pelletization of Iron Ore by Partial or Full Substitution of Bentonite by Organic Binders
CANMET, Mineral Sciences Laboratories
Division Report MSL87-102(IR)
July, 1987, Micromedia

Table 2 - Indurated Strength of Pellets at Various Temperatures

Exp.	Extrusion Mix	Indurated Temp. C	Time min.	Strength N	Time min	Strength N
1	0.6% Greek bentonite	1300	10	1436	20	1620
		1250	10	750	20	1416
		1150	10	520	20	784
		1050	10	216	20	477
2	0.3% alginate	1300	10	549	20	750
		1250	10	490	20	722
		1150	10	248	20	405
		1050	10	147	20	209
3	0.3% MC	1300	10	428	20	770
		1250	10	379	20	594
		1150	10	196	20	500
		1050	10	98	20	180
4	0.1% MC 0.3% Greek bentonite	1300	10	1137	20	1375
		1250	10	856	20	1264
		1150	10	549	20	863
		1050	10	196	20	294
5	0.3% MC 1% $CaCO_3$	1300	10	1117	20	1960
		1250	10	869	20	1744
		1150	10	294	20	539
		1050	10	134	20	255
6	0.3% alginate 1% $CaCO_3$	1300	10	1947	20	2081
		1250	10	1774	20	2009
		1150	10	533	20	852
		1050	10	242	20	343
7	0.3% MC* 1% $CaCO_3$	1300	10	2193	20	2921
		1250	10	2080	20	2525
		1150	10	552	20	944
		1050	10	124	20	186
8	0.3% MC+ 1% $CaCO_3$	1300	10	1741	20	2640
		1250	10	2036	20	2653
		1150	10	490	20	1039
		1050	10	265	20	340

* Pellets were prepared with Mt. Wright iron ore concentrate.
+ Pellets were prepared by mixing 50% Mt. Wright and 50% Carol Lake iron ore concentrates.